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December 1962
Report No. 2440
(Summary)
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DEVELOPMENT OF IMPROVED HIGH-STRENGTH PREIMPREGNATED MATERIALS FOR FILAMENT WINDING

Contract NOW 61-0642-c(FBM)



Structural Materials Division



1974

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DEVELOPMENT OF IMPROVED HIGH-STRENGTH
PREIMPREGNATED MATERIALS FOR FILAMENT WINDING

Contract NOw 61-0642-c(FBM)


AEROJET-GENERAL CORPORATION
Azusa, California

FOREWORD

This is a summary report of the activities of the Aerojet-General Corporation, covering the period of 26 June 1961 through 1 November 1962, on Contract Number NOW 61-0642-c (FEM). This contract is under the direct supervision of the Special Projects Office of the Bureau of Weapons, with Mr. H. Bernstein acting as technical monitor.

This program is being conducted by the Materials Engineering Department of the Structural Materials Division, Aerojet-General Corporation, Azusa. Major responsibility for the program resides with Ira Petker. Other significant contributors to the program include Dr. S. Brelant and M. Segimoto.

AEROJET-GENERAL CORPORATION

A handwritten signature in black ink, appearing to read 'S. Brelant', with a stylized flourish at the end.

Dr. S. Brelant, Head
Materials Engineering Dept.
Structural Materials Division

ABSTRACT

This report summarizes the work on the program for the period ending 1 November 1962. Data is included for six experimental lots of roving, both dry and preimpregnated. Two experimental lots of roving are currently under investigation in the laboratory. Completion of the evaluation of these eight lots of roving will complete the Phase I portion of the investigation. A description is given of the redirected Phase II of the program, which will be devoted entirely to a study of the preimpregnation process.

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I. INTRODUCTION

This program is a joint effort of Owens-Corning Fiberglas Corporation, U.S. Polymeric Chemicals, Incorporated, and Aerojet-General Corporation. The purpose of the program is to develop an improved, preimpregnated glass fiber material (pregreg) suitable for use in filament-wound motor cases capable of withstanding tensile strength (fiber-stress) levels of 375,000 to 400,000 psi at room temperature and maintaining at least 75% of the room temperature strength at 300°F.

The original program was to cover an eight-month period and was to be divided into two phases. Phase I was to establish optimum procedures for fabrication, handling and shipping glass roving, and for applying controlled quantities of resin to glass roving materials. Specific tasks on Phase I were: (a) fabrication of eight 100-lb lots of E-glass with HTS sizing by Owens-Corning under controlled, documented conditions; (b) determination by Aerojet of the mechanical properties of each lot of glass; (c) impregnation, by U.S. Polymeric, of each lot of glass with a resin system suitable for 300°F, under controlled, documented conditions; (d) evaluation of each lot of prepeg by Aerojet; and (e) preparation of tentative material and process specifications for high-strength, high-quality prepeg. Phase II was to be a confirmation of the positive results of Phase I, with a larger sampling of material prepared under production conditions.

As reported earlier, results obtained during Phase I caused several changes in the original Phase I work plan, as well as a complete revision of Phase II. The effect of this has been a substantial increase in the program span time, although no additional funding has been required.

The basic changes and revisions to the program may be summarized as follows:

A. Three of the eight original lots of glass to be provided by Owens-Corning could not be fabricated (as originally planned), with improvements to catenary, fuzz content, and shipping protection. No further improvement to these properties was obtained in special production runs over the improvement noted in earlier lots. The following substitutions were made:

1. Lot 6 was changed from improved catenary to a new thread-wound package.
2. Lot 7 was changed from improved damage and moisture protection to DE filaments (408 filaments per end) instead of the standard ECG's (204 filaments per end).
3. Lot 8 was changed from fuzz-content improvement to standard roving with zero ribbonization.

B. Improvements in the strength of production glass roving by incorporating changes which were developed for the first few lots on this program negated the need for Phase II as originally planned. Phase II has been revised to reflect more critical current needs in the preimpregnation process, and its effect upon the processing characteristics of prepreg.

II. PROGRAM STATUS

The revised program is designed to be completed by 26 January, 1963. Phase I is complete with the exception of evaluation of Lots 6 and 8. Lots 6 and 8 are at Aerojet and are currently being analyzed in the laboratory. Materials for the revised Phase II are also at Aerojet including a lot of standard current production HTS/E-glass.

The object of the revised Phase II program will be to obtain a clearer and more quantitative understanding of the effect of preimpregnation processing parameters upon the application characteristics of the prepreg, and improve the current levels of band width and resin content control. The revised Phase II is divided into three main subtasks. A description of each subtask is given

below. It should be noted that most of Phase II will use the Shell 58-68R resin system in place of E-787 type resin. This change was due to the proprietary nature of the E-787 composition. Since Phase II is to be basically a study of the preimpregnation process, it is necessary that the actual composition of the resin be known so that processing effects which are attributable to the resin system would be capable of interpretation. Another important justification for the use of Shell 58-68R is that it is currently being considered for qualification for the first stage of Polaris; therefore, increased knowledge of the characteristics of this system is pertinent to its successful use.

A. SUBTASK I - PROCESS VARIABLE STUDY

Seven variables which effect prepreg processing characteristics will be studied. Five of these variables (tower temperature, running speed, resin accelerator content, and temperature and specific gravity of the impregnating bath) will be varied between impregnation runs, but will be held at a constant nominal level within a given run. Creel tension and package geometry will be varied between rolls of roving within each run, but will be constant within a given roll. A total of 16 runs will be made; approximately 50 lb of prepreg will be produced during each run.

Among the types of information that will be obtained from the work are the following:

1. Definition of the statistical variation of resin content and band width attributable to variation in the roving and processing equipment.
2. Sensitivity of prepreg filament winding characteristics to variation in preimpregnating processing parameters.
3. A definition of both the maximum and minimum degree of polymerization necessary for satisfactory winding.
4. The effect of degree of polymerization on horizontal shear strength, strand strength, volatile content, etc.

B. SUBTASK II - RESIN CONTENT STUDY

An analysis will be made of the variation in resin content of prepreg produced at U.S. Polymeric during the past few months. Included in the analysis will be variation between and within rolls for both standard and zero ribbonization roving. In addition, chambers will be made from high and low resin content prepreg to verify current specification callouts of 17 to 22%. Finally, special devices to apply pressure and to work the roving will be introduced at several points in the impregnating line; these devices will thoroughly wet the roving and minimize variability.

C. SUBTASK III - BAND-WIDTH CONTROL

Current variability in band width of prepreg is greater than is desired for highly reproducible winding patterns. However, the true variation of this property for prepreg held under tension has not been established. In this study, a procedure will be developed for measuring the band width of roving which is tensioned and the dependence, if any, of band width upon the various parameters used in producing prepreg will be established in Subtask I. The amount of variation, both between and within rolls of prepreg, will be established.

In addition to being potentially dependent on prepreg processing parameters, band width is also dependent upon the roving used to produce the prepreg. The particular characteristics of interest in this respect are the ribbonization of the roving, the degree of twist or crossover, and the degree of bond between ends. In this study both standard roving with high ribbonization and zero ribbonization roving (no bond between ends) will be used. Additional information will be obtained from Lot 6, which consists of zero-ribbonization roving formed onto a parallel-wound package; Lot 6 production methods incorporate more positive means (in the roving forming equipment) to limit crossover and twist than is used in forming the standard way-wind package.

In addition, positive devices to control band width will be studied. This will include passing the "B-staged" prepreg through slotted dies, and over heated pins, which spread the prepreg band. Work on forming a wider prepreg

band by passing the roving over a heated pin was initiated during Phase I. An attempt will be made to optimize the wider band in terms of minimizing variability at the maximum controllable width.

III. TEST RESULTS - PHASE I, SUMMARY

Laboratory test data for all material evaluated is summarized in Table 1 for dry glass, and Table 2 for prepreg. Data from 18-in.-dia chambers is summarized in Table 3. As shown in Table 1, the average lot strength of vinyl-coated strands for all lots, except Lot 2, was between 335,000 and 348,000 psi. The average lot NOL-ring strength, for all except Lot 2, was between 319,000 and 334,000 psi. Thus the lot average range in both tests was less than 5%, indicating little, if any, significant difference in strength between lots. In both tests and range of values for individual rolls of roving was significantly higher, between about 300,000 and 370,000 psi for strands, and between 290,000 and 350,000 psi for NOL rings. Sizing content, as measured by ignition loss, tends to be quite consistent with a nominal average of about 1.45%. A definite improvement in this property over the control lot is demonstrated in the experimental lots. This is particularly true of Lots 1, 3, 4, and 5.

As shown in Table 2, the lot average range for prepreg strands was between 304,000 and 340,000 psi which is a considerably higher spread than for the vinyl strands. Also, the nominal average strength is in general somewhat lower than vinyl strand strength for equivalent lots. However, prepreg NOL rings were very consistent from lot to lot, and also tended to be stronger than the equivalent in-process rings. With the exception of Lot 3, the lot average range for prepreg NOL rings was 336,000 to 349,000 psi which is essentially the same as the range for vinyl strands and in-process NOL rings. The prepreg gravimetric data tends to vary somewhat more than is desirable, although in respect to resin content, Lot 7 (DE filaments) had an unusually small total variation between rolls, a variation of about 1%.

The data from the 18-in.-dia chamber tests, summarized in Table 3, does not indicate any consistent trends. Several chambers, particularly those prepared

from Lots 3 and 5 prepreg, have burst at hoop filament stresses greater than 330,000 psi. However, other chambers have had ultimate hoop filament stresses as low as 260,000 psi.

IV. TECHNICAL DISCUSSION

A. GLASS STRENGTH

The average glass strength measured, for almost all the experimental lots of glass, appears to be relatively constant for any one test method and particular type of material. However, certain inconsistencies appear when the strength data is compared between dry glass and prepreg material, and from one test to another. The prepreg strand strength is consistently lower (by about 8%) than the vinyl-coated strand strength, whereas the prepreg NOL ring strength is consistently higher (by about 5%) than in-process NOL ring strength. Other anomalies appear when the data is subjected to additional analysis. It was found that the vinyl strand strength was higher in general than in-process NOL ring strength. However, prepreg strand strength is consistently lower than prepreg NOL ring strength. Finally for certain lots, including Lots 2, 4, 5, and 7, the prepreg NOL ring strength is equal to or higher than the vinyl strand strength measured for the same glass prior to preimpregnation. Since strand tests, NOL ring tests, and 18-in. chamber tests are the criteria used to evaluate improvements in the prepreg, a discussion and interpretation of this data at this point appears warranted.

The strand test is essentially a test of pure tension, whereas NOL ring testing, by its nature, imposes bending and shear forces which will cause glass failure at a lower stress level than the same glass fibers in pure tension. If the hypotheses are made that the glass fibers are strongest when stressed in pure tension, and that the preimpregnation process may or may not lower, but certainly will not improve the glass strength, the data can be interpreted more meaningfully. First, the highest glass strengths within any given lot of roving should have been for the vinyl strands; these values could be expected to be significantly higher than the corresponding NOL ring strengths. Since this was not true in several

instances, it can be concluded that the vinyl strand test does not stress the glass to its ultimate capability; actual strength of the glass, therefore, is probably higher than is indicated by the test data, and the strand test data should be viewed as defining minimum rather than ultimate strength. The inability of the vinyl strand tested to approach ultimate glass capabilities, is probably associated with the properties of the vinyl resin and with the methods of gripping. A resin which has better wetting characteristics, higher shear strength, and more efficient shear transfer characteristics than the vinyl resin employed (i.e., the same type of resin used in chamber fabrication) would probably produce higher calculated glass stresses. Also, an improved method for distributing the load in the grip area more uniformly would also reduce borderline grip failures.

Although possible problem areas can readily be identified in the vinyl strand test, there are certain positive features of this test which should also be mentioned. As a quality control test the vinyl strand test is probably the simplest and most reliable method for assessing glass strength on a production basis. The improvements which might be made in the test, as noted previously, add complications and time to specimen preparation, testing, and data evaluation which result in significantly higher costs. Another factor which is very important is that the method used in the AeroROVE test to prepare strands does not tend to remove such defects as catenary, crossover, and twist from the roving if these defects are present. This consideration is sometimes overlooked in proposing refinements for the strand test and the actual condition of the roving tested may be different than the roving which originally was on the spool.

The results obtained with prepreg strands are consistently lower than those obtained with prepreg NOL rings; this further substantiates the comments regarding the strand test. However, the lower strength of the prepreg strand, compared to the vinyl strand, still requires an explanation, since prepreg NOL rings and 18-in. chambers have consistently been equivalent to the higher end-strength than in-process NOL rings and chambers made with the same glass. It is particularly important to emphasize that prepreg strand data is the result of a test of a composite rather than a simple test of glass strength. Although the

amount of resin present in prepreg strands is theoretically great enough to completely impregnate the fibers, there is little excess resin available to make up for localized resin deficiencies, or to prevent sequential failure and peeling at localized fiber breaks. In addition to the low resin content, no pressure is used in preparing specimens; therefore, little effective resin migration takes place which could compensate for local resin inadequacies and which could produce more uniform impregnation. Finally, in the grip area, little excess resin is available to protect the fibers and distribute the loads created by the friction grips. The sum total of these factors can make prepreg strands very sensitive to minor variations in resin distribution, spatial relations between ends, band width, position of filament flaws, etc. Indeed, roving impregnated in the laboratory, using the same resin systems, has yielded prepreg strand data which is equivalent to or higher than the vinyl strands.

In the vinyl strand test the vinyl resin not only allows shear transfer between fibers and distributes gripping loads, but also may act as an encapsulant which prevents sequential failure due to peel. A simplified diagram of a postulated mechanism of sequential failure is shown in Figure 1; in the figure, a series of filaments (F_1, F_2, F_3, F_4, F_5) are shown with potential failure sites or defects at locations I, II, and III. F_1 and F_5 are intended to represent filaments on the outside surface of the strand, whereas F_2, F_3 , and F_4 are assumed to be in the interior and completely surrounded by resin and other filaments. Assuming equal severity of defects, it is probable that the initial fiber failure will occur at location I since this fiber has a smaller number of fibers in intimate proximity with which to distribute the load around the defect. Once failure occurs, there is a tendency to peel back as shown in Figure 1b. The phenomenon of peeling has been observed on numerous occasions during strand testing. Due to the filament break and resultant peel, not only is all of that part of the load originally carried by this fiber forced into the remaining intact fibers, but a new filament or group of filaments now occupy the outside surface. If the new outside filament, F_2 in the diagram, has a weak site which is exposed due to peel failure occurs as shown in Figure 1c. Thus a series of events may occur which produces sequential failure until the remaining intact fibers are at a stress level near ultimate and catastrophic failure occurs.

The role of the encapsulant can be explained rather simply and is shown in Figure 1d, and 1e, as a heavy resin coating. In this case when a filament break occurs at the outside at location I, fiber F_1 is restrained from peeling by the "hoop" forces exerted by the resin surrounding it. Therefore, the flaws at locations II and III are essentially "unaware" that failure has occurred in an adjacent fiber and the stress situation in this vicinity is relatively unchanged. As a result, the strand continues to pick up load until catastrophic failure occurs throughout the strand.

Some preliminary work has been done with vinyl-coated prepreg strands, the results of which are shown in Table 4. All these strands were prepared in exactly the same manner as standard prepreg strands except that the prepreg was coated with vinyl resin prior to oven cure. In each case, the vinyl coating increased the failure stress significantly; the most dramatic effect was noted on the strands which originally failed at the lower stress levels. Although this data does not prove the effect of encapsulation, it does tend to support the argument given above.

Another interesting comparison was made between prepreg and in-process NOL rings; it was found that the prepreg rings were consistently higher in strength than in-process rings for the same lots of glass. Based on the original hypothesis that preimpregnation cannot improve glass strength, the explanation for the strength improvement must be found in factors other than glass strength. Although no quantitative analysis is possible at this time, it would appear that the differences would be related to composite differences such as resin content, void content, interfacial shear, etc., or to resin differences such as shear strength, wettability, rigidity, etc. Since ring testing appears to be sensitive to one or more of these factors and it does not measure a true physical property characteristic of any individual component of the composite, caution must be exercised in attributing differences in NOL ring strength to differences in the glass strength.

B. GRAVIMETRIC DATA

1. Roving

Ignition loss (or sizing content) for all experimental lots of material, except Lot 2, has been very consistent both between lots and between rolls within a given lot; this represents an improvement over the control lot. (Lot 7 is an exception but this is probably due to the fact that Lot 7 represented the first DE filaments treated with HTS-type finish.) This improvement is due, at least in part, to the practice of discarding a portion of the roving from the inside and outside of each cake package in which sizing content is most variable. The variability at these positions is due in part to migration effects; these effects occur while the size is liquid, prior to and during the oven bakeout cycle of the cake packages. Volatile losses are probably highest on the outside of the cake packages, since that surface is in direct contact with heated air during the oven cycle.

In order to reduce the variability caused by these effects, a portion of the roving from each cake package is removed from both the inside and outside of each cake. The amount of material removed is based on experimental curves of ignition loss versus position in the cake. Although these curves have been requested, they have not been released by Owens-Corning.

As with ignition loss, weight per lineal yard has been very consistent for all lots of roving. The maximum weight range within any given experimental lot has been 0.015 g. This represents a 50% improvement over the control lot for which a range of over 0.030 g was measured.

2. Prepreg

For the impregnation of all experimental lots of material, no deviation was allowed in the basic impregnation process except as required to maintain resin content at $19 \pm 2\%$. The main impregnation processing parameters and their average levels were:

<u>Parameter</u>	<u>Average Level</u>
Tower temperature	$360 \pm 10^{\circ}\text{F}$
Processing speed	$65 \pm 10 \text{ ft/min}$
Creel tension	750 g
Resin-gel time	$4 \pm 0.25 \text{ min}$

The main parameters which did require adjustment between lots were the specific gravity of the resin bath and creel tension. In the U.S. Polymeric preimpregnation process resin bath specific gravity is the most influential factor in the determination of prepreg resin content. As shown in Figure 2, which shows the approximate effect of solution specific gravity upon prepreg resin content, a change of 0.5% in specific gravity causes a 1.0% change in resin content. Figure 2 is representative of standard 20 E HTS roving, with a ribbonization of 2 or 3, and assumes constant resin pickup characteristics for the roving.

The influence of creel tension can be explained by reference to Figure 3 which is a schematic diagram of the U.S. Polymeric preimpregnation system. As shown in Figure 3, there are only two contact points at A and B prior to Oven 1. At A and B, work is applied to the roving and the strand will tend to spread and break down into individual ends. Roving surface area will become larger as the number of unbonded ends becomes larger and other things being equal, the resin content will also become greater, since it is dependent upon the surface area of roving exposed to the resin in the impregnation bath. Therefore, resin pickup will vary directly with the amount of debonding that occurs at points A and B. The effect of creel tension is that, as it is raised, a greater amount of work will be applied to the roving at these contact points and, therefore, a greater amount of debonding can occur. To sum up, resin content will be dependent upon roving surface area at time of impregnation; this, in turn, is a function of degree of end to end bond, number, and type of contact points and creel tension.

U.S. Polymeric has indicated that variability in end-to-end bond is the most important single roving characteristic responsible for variability in resin content. End-to-end bond may be defined as the amount of work required to break a single 20-end strand of roving into 20 single-end strands. If this property is variable, it is possible that during preimpregnation the degree of end-to-end bond breakdown will also be variable. As noted previously, little work is applied to the roving in the U.S. Polymeric process. The basis for eliminating work is the assumption that under these conditions the minimum amount

of damage will be done to the roving. However, since it is impossible to eliminate all work from the process, debonding does occur for weakly bonded roving. It does not occur for strongly bonded roving. At present there is no method for measuring end-to-end bond and therefore no quantitative analysis of the effects of this property is possible. (The Owens-Corning ribbonization measurement does not consider end-to-end bond since no method is incorporated in the test to apply a controlled and reproducible amount of work to the roving.)

Zero-ribbonization roving has been suggested as one means of eliminating the end-to-end bond problem since this type of roving, having no end-to-end bond, would have a controllable and reproducible surface area. However, it is important to note that any degree of ribbonization is acceptable from the standpoint of resin content uniformity if both the end-to-end bond and the work applied during the impregnation process are both controllable and reproducible. Preliminary experience with zero-ribbonization roving has indicated that the potential improvement in resin content control with this roving may be superseded by other less desirable characteristics. It appears that there is a tendency for catenary and twist buildup to occur during impregnation of zero-ribbonization roving. Normally, these effects are resisted by the end-to-end bond in standard roving. It has been noted that there is a rather high degree of twist and cross-over in zero-ribbonization roving and an apparent lack of it in standard roving. Since the roving forming process is the same for both materials, it would appear that the end-to-end bond in standard roving simply covers up these defects rather than being defect-free as is normally assumed.

An alternative to zero-ribbonization roving would be to apply sufficient work to the roving during impregnation to overcome the maximum degree of end-to-end bond that might exist in the input roving. The potential damage to the roving would have to be considered in any attempt to solve the problem in this manner. However, both this approach, as well as zero-ribbonization roving, are being investigated in the current program.

Thus far, two parameters which effect resin content have been discussed. One factor, specific gravity, is related to the preimpregnation process; the other factor, end-to-end bond, is related basically to the roving but is also

effected by the preimpregnation process. Two other roving properties, which affect resin content and which can be treated quantitatively, are weight per yard and sizing content. However, as will be shown, these properties explain only a small part of the resin content variation of prepreg. If a constant resin pickup is assumed, a variation of ± 0.03 g ($\pm 5\%$) per yard can account for a resin content variation of about $\pm 0.6\%$. However, as was noted previously, the total range of weight per yard in most of the experimental lots of roving was only 0.015 g, whereas the range in resin content was 0.9% for the best lot, Lot 7, and between 2 and 3.5% for all the other lots. It should also be noted that extreme care could be exercised in the impregnation of the experimental roving since only one or two rolls of roving were coated at any one time, compared to as many as 50 rolls under standard production conditions.

The contribution of sizing content to resin content variation is even smaller since a variation of $\pm 0.5\%$ in sizing content can cause a variation of only $\pm 0.06\%$ in resin content. Actually Lot 7 which had the lowest resin content variation had one of the highest sizing content variations of all the experimental lots of roving. Therefore, quantity of sizing and its variability is not a factor of major importance.

Other roving properties which may affect resin content are twist and crossover, affinity of the resin to the finish (wettability), degree of bond between filaments within an end, and degree of penetration of the finish by the resin. There may be other properties which might even be more important than any of those suggested; however, the important factor for all the properties in this group is that no quantitative value can be assigned to any of them at this time and therefore no quantitative analysis is possible. That current quality control at Owens-Corning does not measure a property directly related to resin pickup characteristic, is indicated by the data in Table 5; this table shows the lot averages, the average mean deviation, and the high and low value for the standard quality control tests that are currently applied by Owens-Corning to 20-E-HTS roving. This data is for the experimental roving supplied for this program. At least one measurement of each property was made for every roll of

roving. In addition to these properties, measurements of catenary, ribbonization, and yardage per pound were also made by Owens-Corning. Catenary and ribbonization data are not included in Table 5 because Owens-Corning data indicated that the former property was invariant at a level of zero and that the latter property was invariant at a level of 2 or 3. (Yardage per pound is essentially the same as weight per yard, which was discussed previously.)

Although several of the properties shown in Table 5 may be eliminated a priori as being related to resin pickup, such properties as wetout rate, stiffness, solids content and volatile content could conceivably affect prepreg resin content. However, when these properties are compared to the prepreg resin content data also shown in Table 5, no correlation or trend is apparent.

C. CHAMBER DATA

Although in general the hoop filament stress at burst of the 18-in.-dia chambers has been relatively high, few of the chambers have duplicated the high glass stresses of the simple tensile tests. In the fabrication of an 18-in.-dia chamber, there are a number of variables which do not have a major effect on simple tensile testing but which can affect chamber performance. Filament alignment, winding pattern, tension uniformity, resin flow, air occlusion and mandrel contour are just a sampling of the parameters which can adversely effect chamber burst strength. Theoretically the strength in pure tension of the input roving should be the maximum burst stress that can be expected of a chamber and the difference between this value and the filament stress at burst should indicate the efficiency of the fabrication process. In Figures 4 and 5, chamber filament stress at failure is shown in terms of efficiency factors based on input glass strength. In Figure 4, the efficiency factor was obtained by dividing ultimate hoop filament stress by lot average NOL ring strength. In Figure 5 the efficiency factor used is ultimate hoop filament stress divided by Aerorove strand strength. It is apparent from Figures 4 and 5 that over 80% of the chambers failed at efficiency factors of 0.85 or higher. Although these figures do not indicate a perfect correlation between ultimate chamber filament stress and either strand

or NOL-ring strength, it does point out those chambers for which a serious processing deviation probably occurred. In a positive sense, the figures also indicate that at least 85% of the NOL-ring or strand strength can be expected in a biaxially loaded chamber.

V. CONCLUSIONS

The study to date has produced the following significant conclusions:

A. The Lot 1 improved package has resulted in a higher average dry glass strength. This conclusion is substantiated by the strength of current production prepreg. As shown in Table 6, the average lot strength of production prepreg for the period between June 1962 and September 1962 was between 367,000 and 400,000 psi. Just prior to this period the lot average strength was about 340,000 psi. These values indicate a significant upward trend, especially when compared to an average prepreg strand strength of between 260,000 and 300,000 psi for production prepreg received less than one and one-half years ago. The original contention of this program that improvements to the roving forming process and more intensive quality control procedures would recover a major fraction of the virgin glass strength appears to be validated by the strength of current production roving.

B. The preimpregnation process does not materially damage roving, but does result in a fabrication material which produces composites equivalent to or better than those produced by the in-process or wet impregnation process.

C. Current methods for measuring the tensile strength of glass in a composite require modification and optimization before truly quantitative analysis can be made of the efficiency of chamber processing, design and the influence of resin properties.

D. A method for measuring the resin pickup characteristics of roving is highly desirable in order to guide the development of prepreg with improved resin content control and wetout characteristics. There are several roving properties of importance in this regard and probably each should be treated independently. Of major importance would be methods to measure end-to-end bond, twist-and-crossover, and the variability of finish to wetting.

E. Zero ribbonization roving which is currently under analysis would probably permit improved control of prepreg resin content and bond width. However, to be used successfully, twist and crossover would probably have to be eliminated from the roving and careful control would be necessary during impregnation in order not to introduce fiber misalignment.

F. Preliminary work with DE filaments has indicated a potential improvement to be obtained from their use. Although there appears to be no strength advantage in filaments of smaller diameter, resin-content control and composite uniformity may be improved.

TABLE 1

GRAVIMETRIC AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING

[illegible]

TABLE 2
GRAVIMETRIC AND STRENGTH DATA OF EXPERIMENTAL PREPREG

Lab. No.	37-2758 (Control)	1-2794	2-2807	3-2808	4-2826	5-2828	7-2842
Lab. No.							
Date							
Operator							

VELOCITY TENSILE STRENGTH (STRAINS)	
Average in No. 1	Ave 303.7 Ave 307.0 Ave 322.7 Ave 321.8 Ave 339.4

TENSILE STRENGTH (LOAD ON TENSION)	
Percent	Ave 18.72 Ave 18.15 Ave 19.36 Ave 20.16

VOLATILE CONTENT	
Percent	Ave 1.54 Ave 1.06 Ave 1.94 Ave 1.76 Ave 1.81 Ave 1.68

VOL. RES. (GLASS STRAINS)	
Average in No. 1	Ave 336.1 Ave 338.9 Ave 335.8 Ave 343.3

TABLE 3

18-IN. DIAMETER FILAMENT WOUND CHAMBERS - TEST RESULTS

Chamber Number	Material	Burst Pressure (psi)	Hoop Filament Stress-Cylinder (ksi)	Long. Filament Stress-Cylinder (ksi)	Composite Stress-Cylinder (ksi)	Long. Composite Stress-Cylinder (ksi)	Long. Composite Stress-Head (ksi)	Strander Strength (ksi)	ROL Ring Strength (ksi)
0090-163	Lot 37-400	866***	305.9	245.5	127.2	63.6	165.7	297.0	-
0090-164	Lot 37-M	895	311.2	235.2	126.6	63.3	158.8	337.0	-
0525-3	Lot 1-M	860	267.1	226.2	113.4	56.7	152.7	363.7	320.5
0525-4	Lot 37-P	740	260.0	200.2	104.2	52.1	132.2	333.3	323.0
0525-5	Lot 1-P	950	297.8	236.3	120.8	60.4	156.2	311.1	337.1
0525-6	Lot 3-M	910	306.9	231.6	124.4	62.2	156.1	333.3	297.8
0525-7	Lot 2-M	960	322.1	249.7	132.2	66.1	168.5	285.3	323.4
0525-8	Lot 4-M	890	294.1	235.8	121.0	60.5	159.2	344.4	324.0
0525-9	Lot 5-M	870	307.8	212.8	120.6	60.3	143.6	355.7	321.0
0525-10	Lot 3-P	930	333.3	232.4	126.8	63.4	153.6	338.0	318.5
0525-11	Lot 2-P	900	297.7	232.8	118.8	59.4	153.9	292.2	344.9
0525-12	Lot 4-P	852	280.1	235.0	112.9	56.5	148.7	316.4	333.8
0525-13	Lot 4-P	790	267.4	208.0	106.4	53.2	137.5	324.9	327.7
0525-14	Lot 5-P	980	337.8	251.3	132.1	66.6	166.1	342.5	342.7
0525-15	Lot 7-M	875	287.1	216.7	115.3	57.7	146.3	327.9	331.7
0525-17	Lot 7-P	900	303.1	234.5	118.6	59.3	152.7	341.4	345.1

* Values are for roll of roving from which chamber was made.

** W = wet winding; P = prepreg.

*** All chambers were weak hoop and failed in the hoop wraps. All chambers were CBO design. Test pressure prior to burst was 400 psi for one minute.

TABLE 4
EFFECT OF VINYL COATING ON PREPREG STRAND STRENGTH

<u>Lot No.</u>	<u>Roll No.</u>	<u>Ultimate Tensile Strength (psi x 10⁻³)</u>					
		<u>Standard Prepreg Strand</u>		<u>Vinyl Coated Prepreg Strand</u>		<u>Vinyl Coated Prepreg Strand</u>	
		<u>Average</u>	<u>High</u>	<u>Average</u>	<u>High</u>	<u>Low</u>	<u>Low</u>
3-804	19	293.4	310.2	377.2	391.2	352.0	
4-826	7	324.8	358.9	393.1	406.3	371.5	
5-838	12	345.4	353.0	381.2	408.6	338.0	
7-846	7	353.1	365.8	399.7	412.0	377.3	

TABLE 5

OWENS-CORNING QUALITY CONTROL
DATA FOR EXPERIMENTAL GLASS

Lot No.	Stiffness	Volatile Content (%)	Solids Content (%)	Wet-Out Rate	Package Hardness	Fuss Content (x10)	Resin Content (%)		
							Avg.	High	Low
1	3.88±0.11	0.204±0.010	1.12±0.03	89.50±1.50	66.1±3.5	0.08±0.045	20.51±0.84	21.82	19.47
2	3.20±0.11	0.015±0.004	1.21±0.07	89.70±1.48	68.6±1.7	1.54±0.65	18.98±0.85	20.80	17.20
3	3.57±0.07	0.106±0.015	1.19±0.03	86.76±2.16	69.2±1.7	1.64±0.60	19.83±0.95	22.17	18.50
4	3.26±0.19	0.246±0.017	1.06±0.03	85.89±1.33	74.5±1.7	2.75±0.50	18.21±1.28	19.92	15.01
5	3.49±0.11	0.106±0.035	1.23±0.04	89.07±1.67	69.3±1.6	1.88±0.38	19.39±0.33	20.40	18.58
7	2.98±0.08	0.165±0.018	1.30±0.04	92.38±1.88	74.3±2.8	4.60±0.50	20.10±0.26	20.58	19.67

TABLE 6

ULTIMATE TENSILE STRENGTH OF PRODUCTION PREPREG

<u>Date</u>	<u>Lot No.</u>	<u>No. Rolls Tested</u>	<u>Strand Tensile Strength(psi)</u>		
			<u>Average</u>	<u>High</u>	<u>Low</u>
April 1962	89/F911	13	337	359	298
April 1962	90/F911	13	342	353	319
May 1962	91/F917	15	338	349	306
May 1962	92/F923	26	346	372	310
May 1962	94/F935	13	362	400	329
June 1962	95/F942	15	400	419	370
June 1962	96/F944	15	396	405	376
June 1962	97/F950	15	373	388	335
July 1962	100/F961	15	383	406	328
July 1962	101/F965	15	382	405	353
July 1962	103/F973	15	378	402	352
July 1962	105/F971	15	374	399	353
July 1962	107/F979	14	376	414	360
August 1962	106/F976	15	381	396	352
August 1962	108/F983	15	367	390	339

ENCAPSULATION EFFECT AND SEQUENTIAL FAILURE OF STRANDS

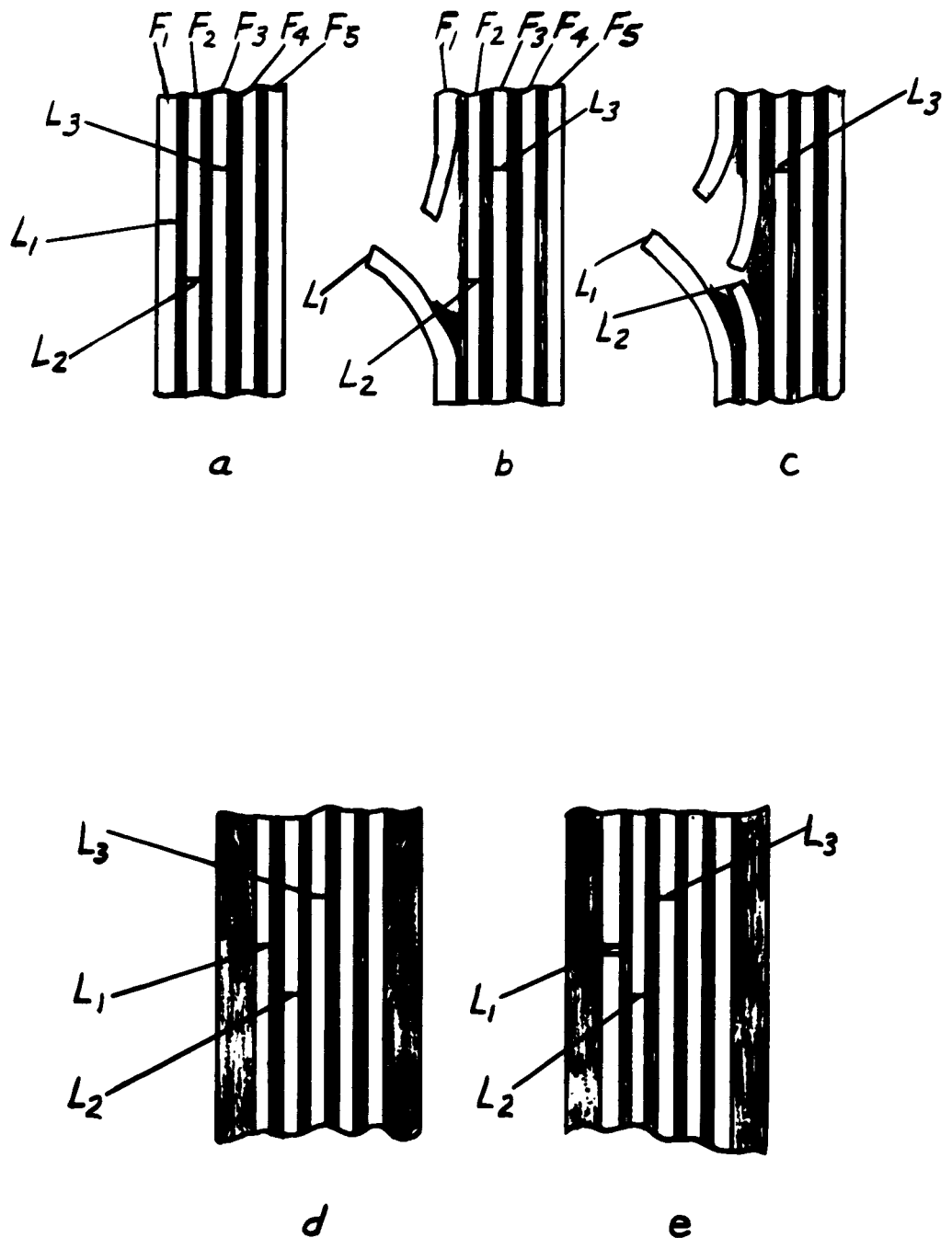


Figure 1

SPECIFIC GRAVITY OF RESIN IMPREGNATING BATH
VS RESIN CONTENT OF PREPREG

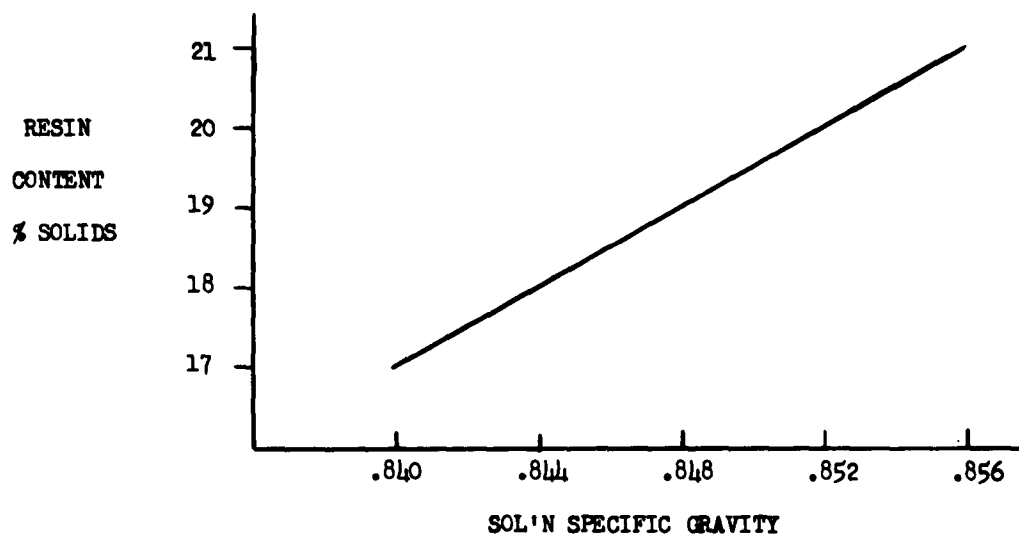


Fig. 2

SCHEMATIC DIAGRAM OF U.S. POLYMERIC PREIMPREGNATION LINE

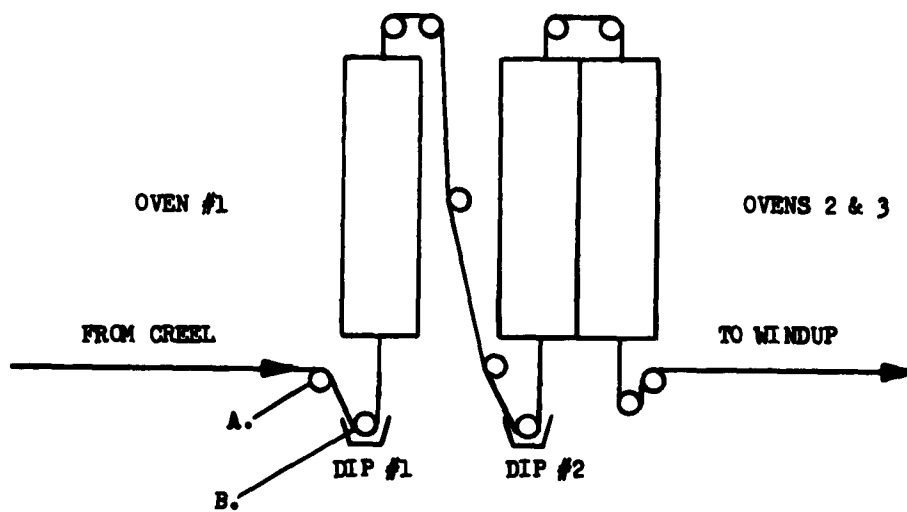
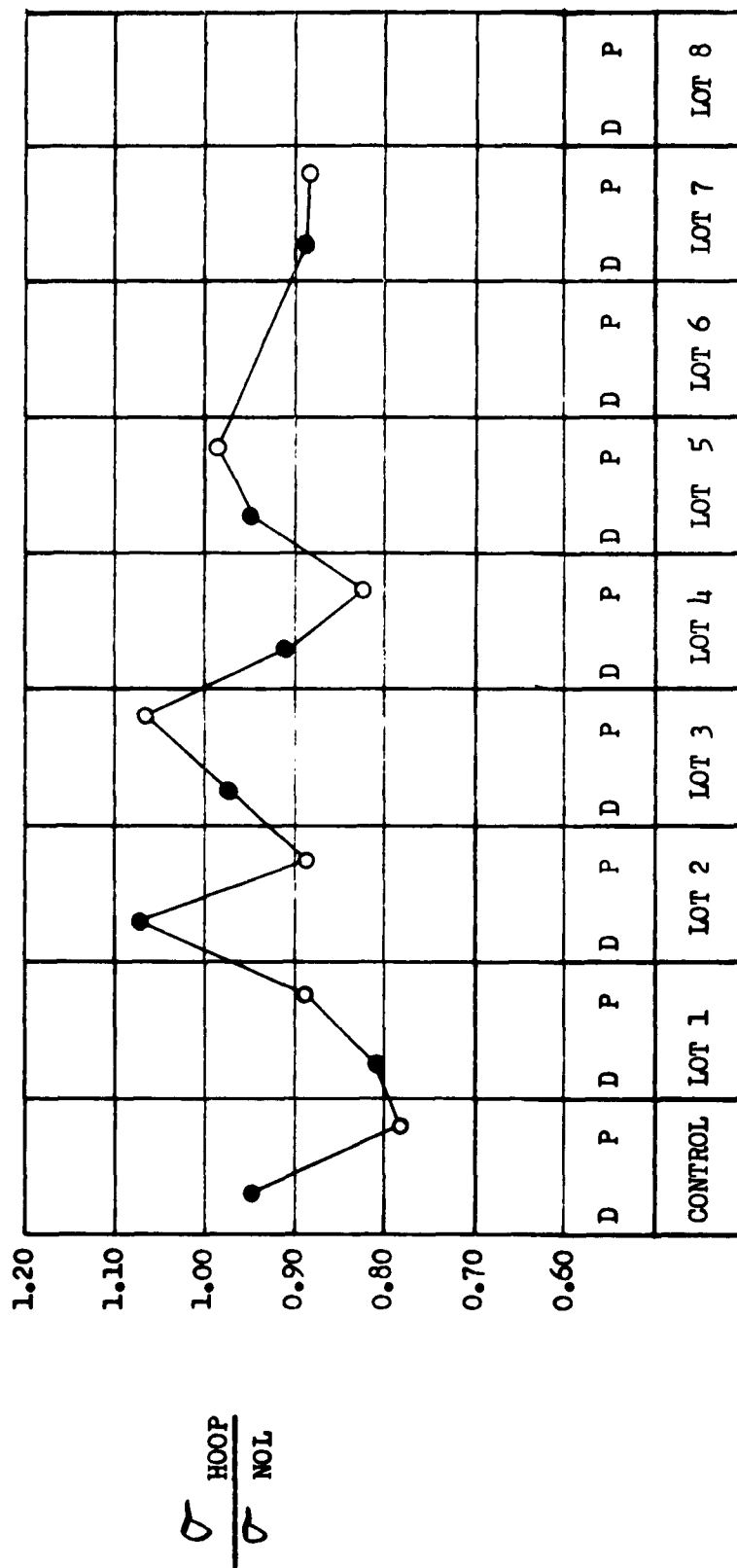
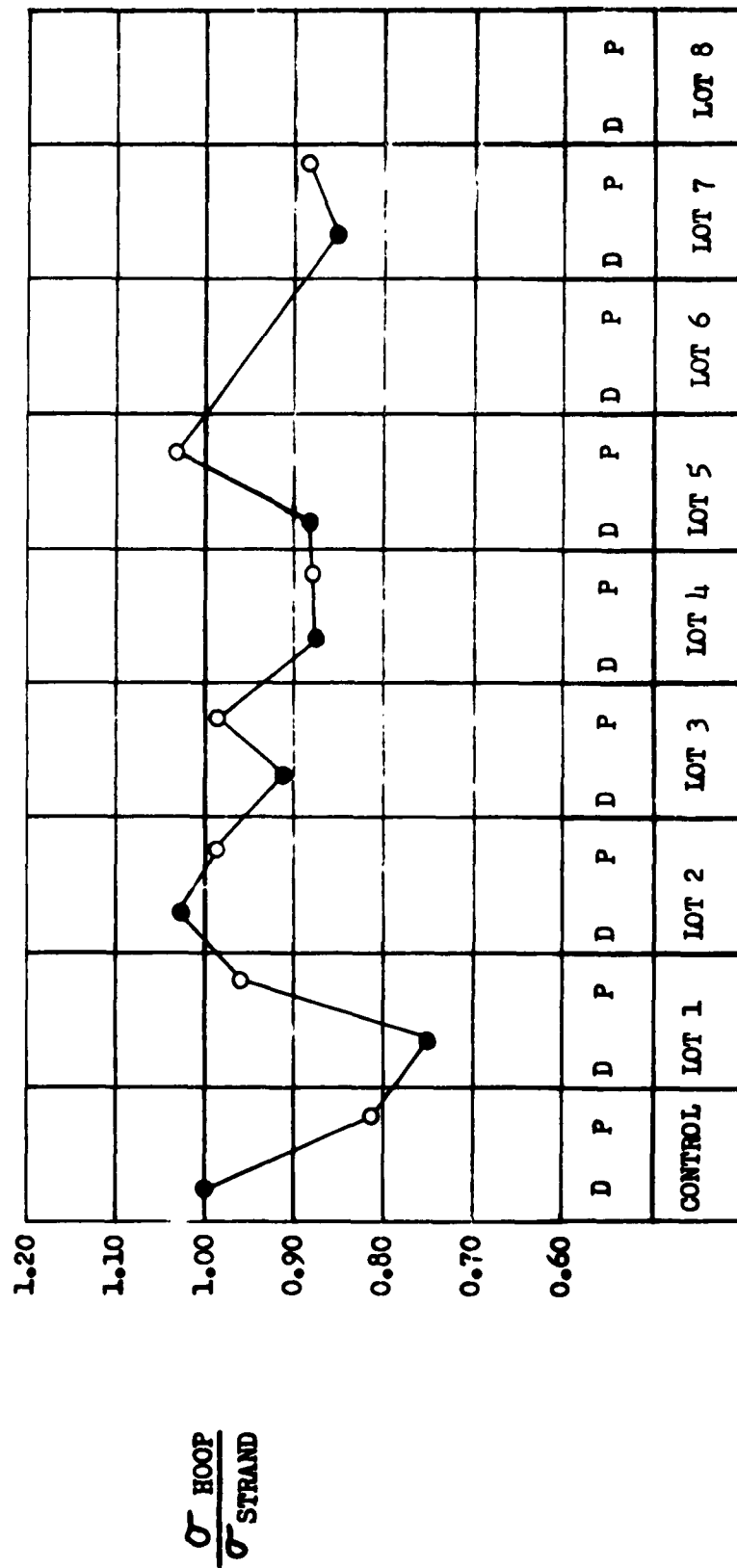


Fig. 3



Comparison of NOL Ring Data and Chamber Data
(D indicates dry; P indicates prepreg.)



Comparison of Strand Data with Chamber Data.
(D indicates dry; P indicates prepreg.)

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